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REVIEW AND RESEARCH ON CARBON FIBER REINFORCED
QUARTZ USED AS THERMAL PROTECTION SHIELD

by

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ABSTRACT An evaluation was done of the characteristics of fiber reinforced ceramics in radiation thermal protective structures acting as exterior radiation cover plates. Using as a basis typical mechanical environments and thermal environments associated with returnable spacecraft, experimental research was carried out with regard to carbon fiber reinforced quartz--including such items as small plate reentry thermal simulation tests as well as room temperature vibration, room temperature shock, low temperature vibration, and low temperature shock associated with large scale thermal structural members, along with reentry heating, and so on. A series of tests clearly demonstrated that carbon fiber reinforced quartz not only retained ceramic resistance to high temperatures and good insulation characteristics, but also overcame inherent ceramic brittleness. As a result, it possesses considerable potential to act as thermal protective material on returnable spacecraft.

KEY WORDS Thermal protective device Quartz Carbon fiber reinforced composite Research

1 INTRODUCTION

Ever since the introduction of returnable spacecraft to the world, radiation thermal protection has right along been a type of thermal protective technology with broad applications. The reason is that this type of thermal protective structure possesses other forms of incomparable advantages. On the outside of protective structures, covering plates capable of withstanding high temperatures are installed. In conjunction with this, covering plate surfaces are made to possess high radiation rate characteristics. Then, in reentry environments, a large part of aerodynamic heating on returnable spacecraft surfaces will be reflected into space by reradiation methods, thereby effectively protecting interior structures.

On the basis of radiant thermal protective mechanisms [1], radiation thermal protection is a physical process which does not generate loss of materials. As a result, it possesses the characteristics that follow. (1) Qualities of thermally protected structures do not depend on overall amounts of heat added. Therefore, however long heating periods are and however large the amounts of heat added are, there is, however, no need to make thermal protective layers heavier. (2) During reentry processes, the exterior shapes of thermal protection surfaces do not change. (3) They possess a potential for reuse. These characteristics are all difficult to produce in other forms of thermal protection. With regard to these low heat flow densities and long reentry periods, it is necessary that exterior shapes not change in order to maintain control of lift forces associated with returnable spacecraft during reentry. Radiation thermal protective structures are the most appropriate.

In radiation thermal protective structures, as far as the highest operating temperatures associated with covering plates are concerned, it is possible to simply make use of radiation equilibrium temperatures in order to do estimates [1]. Radiation equilibrium temperatures are a type of ideal surface temperature.

At these temperatures, all of the aerodynamic heat surfaces undergo is dissipated away in the form of radiation. Any heat transmission process associated with radiant thermal protective structures is capable, in all cases, of being simplified into the models in Fig.1. Fig.1(a) represents surface thermal equilibrium. (b), by contrast, is two types of idealized configuration. At this time, the path of amounts of heat transmitted from cover plates to the interior of structures is completely blocked. Cover plates are in a thermal equilibrium condition. At this time, skin temperature, that is, radiation

equilibrium temperature, is expressed as

$$q=\sigma\varepsilon T4$$

In the equation, q---heat flow density associated with net aerodynamic heating; /62

σ---Boltzmann constant;

ε---surface radiation coefficient;

T---surface thermodynamic temperature.

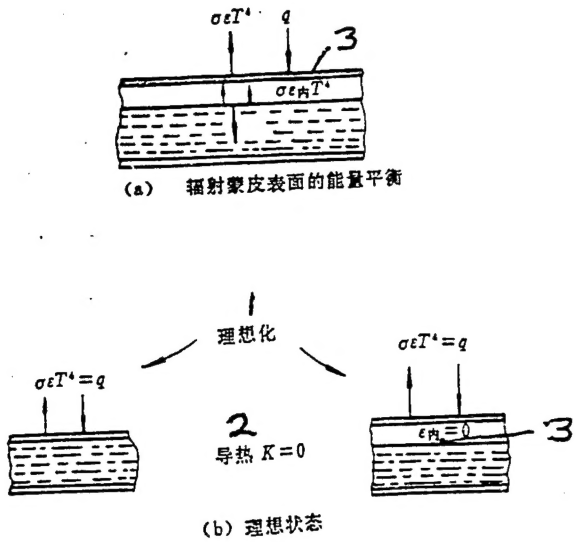

Fig.1 Radiation Thermal Protective Models (a) Radiation Skin Surface Energy Equilibrium (b) Ideal Configurations Key: 1 Idealization 2 Thermal Conductance 3 Interior

Fig.2 is the relationship between radiation equilibrium temperatures and heat flow densities when radiation coefficients are 0.8. In Fig.2, heat flow densities associated with several types of typical returnable spacecraft as well as typical sections are also shown. It can be seen that, when cover plate temperatures reach 3000°C, maximum heat flow densities can reach 3.67x106W/m2 . This heat flow density is larger than all the heat flow densities associated with most of the areas of returnable spacecraft. As a result, it can be seen, from the angle of materials, that radiation thermal protection is capable of application to any section. Fig.3 shows maximum utilization temperatures associated with various types of materials.

Limits on material utilization temperatures are mainly dependent on their high temperature strengths and resistance to oxidation. In this area, ceramic materials possess a natural abundance of these conditions. In various types of materials, ceramic temperature resistance is high. Besides this, ceramic material strengths almost do not vary as a function of temperature. However, other materials--for instance, the

strengths of high temperature alloys and refractory metals--all go down as functions of rises in temperature. Generally speaking, ceramic densities are far smaller than metals. As a result, material strength-density ratios are greater than a good number of high temperature alloys. This type of advantage is even more obvious at high temperatures (see Fig.4).

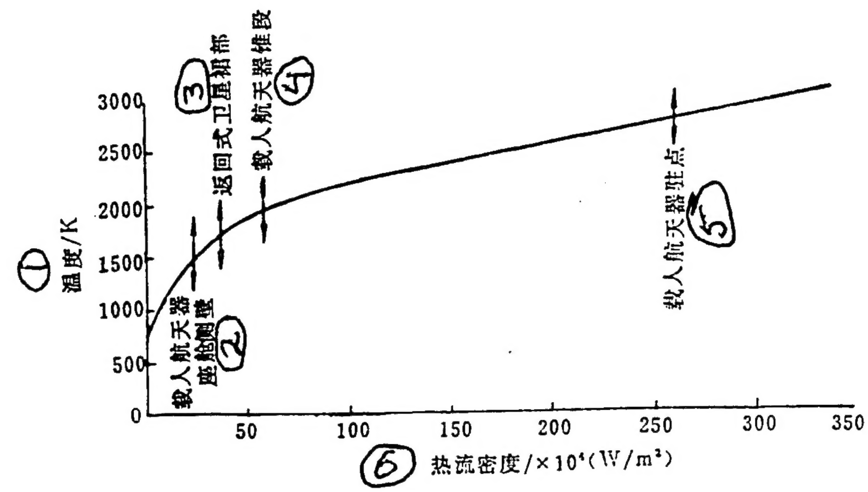

Fig.2 Radiation Skin Operating Temperatures

Key: (1) Temperature (2) Manned Spacecraft Cockpit Side Walls (3) Returnable Type Satellite Skirts (4) Manned Spacecraft Cone Section (5) Manned Spacecraft Stationary Point (6) Heat Flow Density

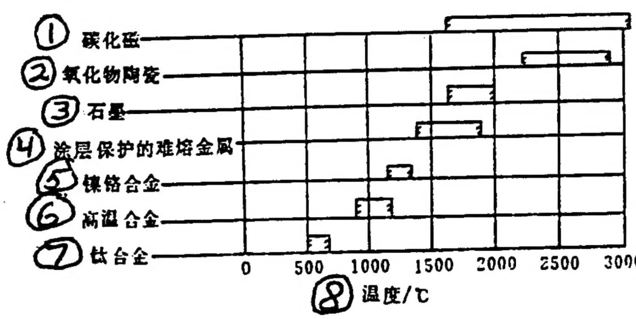

Fig.3 Maximum Utilization Temperatures Associated with Various Types of Materials

Key: (1) Carbonized Magnetic (2) Ceramic Oxides (3) Graphite (4) Coating Protected Refractory Metals (5) Nickel-Chromium Alloy (6) High Temperature Alloy (7) Titanium Alloy (8) Temperature

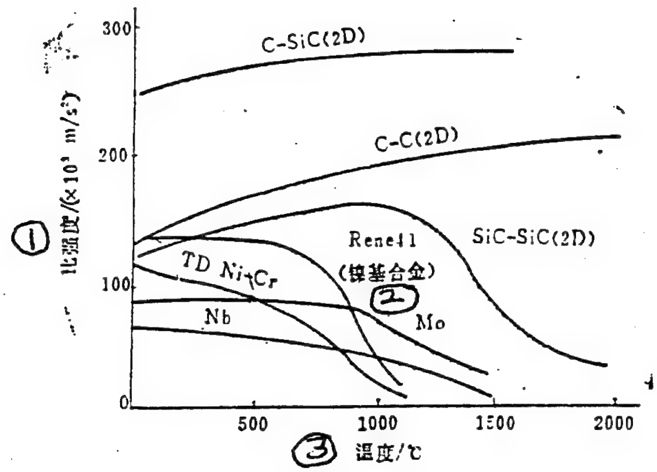

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Fig.4 Changes Associated with Specific Strengths of Several Types of Materials as a Function of Temperature

Key: (1) Specific Strength
(2) Nickel Alloy (3) Temperature

2 RESEARCH AND ESTIMATES ASSOCIATED WITH CARBON FIBER ENHANCED QUARTZ ACTING AS RADIATION THERMAL PROTECTIVE COVERING PLATES

2.1 Investigation of Ceramic Composite Materials

A basic obstacle blocking large numbers of ceramic applications in reentry thermal protective structures is the brittleness of the materials. As far as the inherent characteristics of ceramic materials due to preparation techniques are concerned, there is no way to eliminate the initial microcracks in the interiors of the materials. They are the basic source of destruction associated with ceramic brittleness.

When ceramic products are influenced by external loads, the points of microcracks then produce stress concentrations. Moreover, ceramics, which are brittle in themselves, also lack plastic deformation capabilities in order to relax internal stresses. Therefore, microcracking rapidly expands, finally leading to products producing sudden destruction.

Improving ceramic brittleness has right along been one of the targets which materials scholars have hankered after achieving. Through a great many years of exploration, people discovered that adding certain fiber materials into ceramic bases, the brittleness of the bases will clearly be improved. The Academia Sinica's Shanghai silicate research institute carried out research in this area. Fibers tested included tungsten wire, molybdenum wire, boron fiber, carbon fiber, and carborundum crystal strands. Substrates, by contrast, included aluminum oxide, zirconium oxide, silicon nitride, and quartz glass. Research clearly shows that only when the two fibers and substrates not only possess matching thermal expansion characteristics but also mutual chemical compatibility, only then is it possible to combine materials with good properties. Table 1 sets out the main results of experimental research. Thermal expansion coefficients associated with tungsten wire, molybdenum wire, and boron filaments were far smaller than aluminum oxide and zirconium oxide substrates. As a result, when combined, substrates show the appearance of cracking in directions perpendicular to fibers. Carbon fibers, by contrast, because of radial expansion coefficients being excessively small, lead, when combining them with aluminum oxide and zirconium oxide substrates, to substrates and fibers not being able to combine. Other pairings also--due to fibers and substrates having reactions at high temperatures--are not able to achieve

satisfactory composite materials. However, thermal expansion coefficients associated with carbon fibers and quartz glass are quite close. Moreover, reactions of quartz and carbon at high temperatures are also relatively weak. As a result, it is possible to obtain ideal material composite characteristics.

2.2 Property Estimates for Carbon Fiber Enhanced Quartz

In order to estimate the properties of carbon fiber enhanced quartz (below designated as carbon/quartz), Table 2 sets out partial room temperature mechanical properties. Table 3 sets out partial thermal physical properties. Based on Table 2 and Table 3, it is possible to see that this type of material--besides retaining the normal advantages of ceramics--also has the advantages that follow for acting as radiation thermal protective material.

(1) Densities are low. Specific strengths are high.

Carbon quartz densities associated with two types of industrial techniques both do not exceed 2g/cm3. Compared to refractory metals which can be made use of in the same way at high temperatures, there is a 4.5 fold decrease. As far as carbon/quartz room temperature mechanical properties are concerned, although they are lower than these metals, speaking in terms of basic natures, however, right up to quartz generating/64

phase changes and fusing, carbon/quartz strength losses are not great. However, with respect to the majority of metals--including refractory metals--up to 1200°C and above, strengths have already very, very greatly decreased.

Table 1 Fiber and Ceramic Substrate Composite States

1 纤维	2 基体	3 复合的结果
4 钨丝	5 氧化铝	6 基体在垂直于纤维方向上出现裂纹
7 钽丝	5 氧化铝	6 基体在垂直于纤维方向上出现裂纹
10 硼纤维	5 氧化铝	6 基体在垂直于纤维方向上出现裂纹
4 钨丝	8 氧化锆	6 基体在垂直于纤维方向上出现裂纹
7 钽丝	8 氧化锆	6 基体在垂直于纤维方向上出现裂纹
11 碳纤维	5 氧化铝	13 基体出现严重裂纹，基体与纤维脱离
11 碳纤维	5 氧化锆	13 基体出现严重裂纹，基体与纤维脱离
11 碳纤维	5 氧化锆	14 基体有裂纹，基体与纤维有化学反应
11 碳纤维	9 氮化硅	15 碳和氮化硅在1650°C以上有化学反应
12 碳化硅晶须	9 氮化硅	16 1600°C以上，碳化硅晶须消失
11 碳纤维	2 石英玻璃	17 形成较好的复合材料

Key: (1) Fiber (2) Substrate (3) Combining Results (4) Tungsten Wire (5) Aluminum Oxide (6) Substrate Cracking Appears in Directions Perpendicular to Fibers (7) Tantalum Wire (8) Zirconium Oxide (9) Silicon Nitride (10) Boron Fiber (11) Carbon Fiber (12) Carborundum Crystal Strands (13) Substrate Shows Severe Cracking. Substrate and Fibers Separate. (14) There Is Substrate Cracking. Substrates and Fibers Have Chemical Reactions. (15) Carbon and Silicon Nitride Have Chemical Reactions at 1650°C. (16) Above 1600°C, Carborundum Crystal Strands Disappear. (17) Formation of Relatively Good Composite Materials.

(2) Carbon Fiber Enhancement Overcomes Quartz Brittleness

Comparing carbon quartz to pure quartz glass--in terms of strengths--there are increases of great scope. Bending strengths of carbon/quartz associated with unidirectional fiber arrangements reach 12 times those for pure quartz glass. Moreover, breaking powers characterizing material ductility indices, by contrast, are raised 2-3 orders of magnitude compared to pure quartz glass.

(3) Thermal Expansion Coefficients Extremely Small

Carbon/quartz material thermal expansion coefficients approach expansion coefficients of quartz, that is, $0.69 \times 10^{-8}(^\circ C)^{-1}$ (0-900°C). However, as far as high temperature metals in general are concerned--for instance, thermal expansion coefficients associated with niobium alloys from room temperature to 900°C--they are, by contrast, $(6.5-8) \times 10^{-8}(^\circ C)^{-1}$. As a result, no matter whether it is alternations of high and low temperatures in sections of orbital motion or high temperature conditions associated with reentry processes, thermal stresses on materials are very small. In structural terms, the extension and contraction cracks which remain from resolving high temperature expansion are also very small. For instance, with respect to skins with scales of 700mm, when carbon/quartz is used for manufacturing, the amount of expansion at 1400°C is only 0.63mm. When niobium alloys are used in manufacturing, it then reaches 8mm. In terms of structure, the former will be much easier to realize. What is particularly worth paying attention to is that expansion coefficients of carbon/quartz and quartz are very close. As a result, there are unique advantages in making the mutually embedded parts associated with quartz antenna windows. The two will not produce mutual thermal stresses.

(4) Surface Radiation Coefficients Are High

Radiation coefficients associated with untreated carbon/quartz surfaces are greater than 0.85. Moreover, experiments also clearly demonstrate that radiation coefficients will also go up as a function of surface temperature increases. High radiation coefficients are capable of increasing surface heat scattering, raising thermal protective results.

(5) Thermal Conductance Coefficients Are Small. Specific Heat Capacities Are Large.

Carbon, in a fiber form, dispersed inside quartz, produces very large contact thermal resistance. At the same time, it makes quartz lose transparency at high temperatures, leading to increases in internal radiation thermal resistance. Compared to

refractory metals, thermal conductance coefficients associated with carbon/quartz from room temperature to 1500°C are smaller by approximately 1 order of magnitude. Specific heat capacities, by contrast, are larger by 1 order of magnitude. /65

Table 2 Carbon Quartz Material Room Temperature Mechanical Properties

(1) 项　　目	(2) 碳/石英	(3) 石英玻璃
(4) 密度/[g/cm³]	2.00	2.16
(5) 碳纤维含量/体积%	30	—
(6) 抗弯强度/Pa	5.88×10^{8}; 2.94×10^{8}*	5.05×10^{7}
(7) 抗弯弹性模量/Pa	6.77×10^{10}*	—
(8) 拉伸强度/Pa	5.[illegible]0 $\times10^{7}$*	—
(9) 拉伸弹性模量/Pa	2.58×10^{10}*	—
(10) 泊松比	0.14*	—
(11) 断裂应变/(%)	0.32*	—
(12) 剪切强度/Pa	2.45×10^{7}	—
(13) 冲击强度/(N·m/m²)	4.09×10^{6}	1.02×10^{5}
(14) 断裂功/(J/m²)	7.90×10^{3}	5.94～11.3

(15) 注：本表中含·号是纤维正交排列的材料；其余均为纤维单向排列。

Key: (1) Item (2) Carbon/Quartz (3) Quartz Glass (4) Density (5) Carbon Fiber Content/Volume % (6) Bending Strength/Pa (7) Bending Elasticity Modulus/Pa (8) Tensile Strength/Pa (9) Tensile Elasticity Modulus/Pa (10) Poisson Ratio (11) Rupture Strain/(%) (12) Shear Strength/Pa (13) Shock Strength/(N.m/m2) (14) Rupture Work/(J/m2) (15) Note: The . symbol contained in this table designates materials associated with orthogonally arranged fibers. The rest are all fibers arranged in one direction.

Table 3 Physical Properties of Carbon/Quartz (Fibers Arranged in a Single Direction)

1 项目	2 温度/°C	3 性能指标
4 热膨胀系数/(1/°C)	0～900	0.69×10^{-6}
5 导热系数/〔W/(m·K)〕	500	10.84
	900	16.93
	1 300	23.73
6 比热容/〔J/(kg·K)〕	700	1 306
	1 000	1 402
	1 300	1 482
7 表面半球向全辐射系数	室温 8	0.86
	787	0.78
	1 144	0.70
	加热后 9	0.89

Key: (1) Item (2) Temperature (3) Property Index (4) Thermal Expansion Coefficient (5) Thermal Conductance Coefficient (6) Specific Heat Capacity (7) Full Surface Hemispheric Radiation Coefficient (8) Room Temperature (9) After Heating

2.3 Environmental Simulation Tests Associated with Carbon/Quartz Materials

Estimates of material properties have already fully demonstrated that carbon/quartz clearly possesses very great potential to act as spacecraft reentry thermal protective cover plates. In order to further check whether or not this type of material is capable of acting as thermal protective material for reentry spacecraft. Using typical flight environments associated with China's spacecraft as a foundation--with a view to thermal protection properties and brittleness of materials--mechanical and thermal environment simulation tests were carried out.

(1) Flight Mechanical and Thermal Environments

Thermal protective materials need to withstand mechanical, spacial, and aerodynamic heating environments associated with such various stages as main power stage, orbital motion stage, as well as reentry flight stage, and so on. Speaking in terms of/66 carbon quartz materials, there are a number of environments which certainly are not key. There are those, by contrast, which may be critical to their utilization. During the main power flight phase, before entry into orbit, room temperature vibrations and shocks can be fatal to brittle materials. In orbital flight stages, materials can be placed in extremely low temperature configurations. At the same time, shocks and vibrations created by braking rocket ignition and section separation are also suffered. This will test low temperature material ductility. During reentry flight, thermal protective materials primarily undergo high temperature tests.

Table 4 sets out a vibration and shock environment associated with returnable type satellites for vibration and shock conditions in manned spacecraft at room temperature and low temperatures (-100°C) F, which is similar to this. Fig.5 shows changes in thermal flow densities as a function of time for two typical parts of spacecraft stationary point areas as well as certain nonstationary area points associated with recovery modules for returnable type satellites.

Table 4 Spacecraft Room Temperature and Low Temperature Vibration and Shock Ranges

(1) 振动试验条件	(2)频率范围	15~50Hz (6)		50~2000Hz		
	(3)振动级	1.0mm(半幅)		128		
	(4)方向	平行与垂直结构面板两个方向 (7)				
	(5)试验时间	(8)对数扫描，每个方向半循环(50~2000Hz)15min				
(9) 冲击试验条件	(10)波形(半正弦波)	(11) 加速度幅值 50g	(12)持续时间 6~10min	(13)方向 垂直面板	次数 (14) 2	

Key: (1) Vibration Test Conditions (2) Frequency Range (3) Vibration Level (4) Direction (5) Test Time (6) Half Amplitude (7) Two Directions Associated with Parallel and Perpendicular Structural Surface Plates (8) Logarithmic Scanning. Each Direction Half Cycle (50-2000 Hz) 15 min (9) Shock Test Conditions (10) Wave Forms (Half Sine Wave) (11) Acceleration Amplitude (12) Sustainment Time (13) Direction Perpendicular to Surface Plates (14) Iteration Number

(2) Small Plate Thermal Simulation Test

Before comprehensive environmental tests were carried out on large scale structural plates, use was made of 100mmx100mm square plates on radiant heaters to carry out reentry simulation tests. As far as the tests are concerned, heating was done in accordance with the maximum heat flow density curve in Fig.5. Test samples were 3 types: carbon/quartz with thicknesses of 2-2.5mm, Mo-0.5Ti with thicknesses of 3mm, and Mo-0.5Ti with thicknesses of 0.3mm coated with 1mm layers of Al_2O_3. Main test results are set out in Table 5.

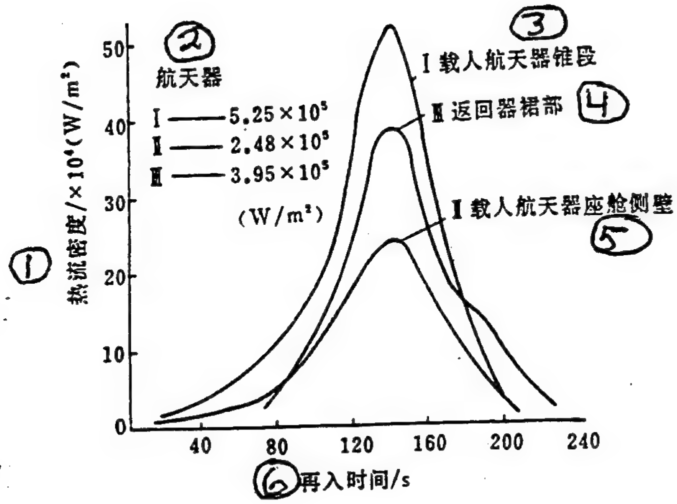

Fig.5 Typical Recoverable Vehicle Reentry Heat Flow Densities

Key: (1) Heat Flow Density (2) Spacecraft (3) Manned Spacecraft Nose Section (4) Returnable Vehicle Skirt Section (5) Manned Spacecraft Cockpit Side Wall (6) Reentry Time

The conclusions below can be obtained from these tests.

1) Carbon/quartz materials are capable of operating at heat flow densities of 5.25x105W/m2 . The heat flow densities in question are maximum values associated with nonstationary point areas. The surface temperatures are far lower than the radiation equilibrium temperature 1534°C associated with $\varepsilon=0.85$. They are also lower than Molybdenum alloy surface temperatures. The explanation for surface temperatures dropping is that carbon/quartz materials at high temperatures possess radiation coefficients greater than 0.85. The intrinsic heat capacities of other materials are relatively large. They also make surface temperatures clearly drop. Seen in terms of amounts of heat absorbed by unit areas of test samples, carbon/quartz materials are 3 times greater than molybdenum alloy. As far as surface /67

temperature drops are concerned, they are advantageous to multiple uses of materials. It is also possible to reduce internal insulation layer quality.

2) As far as carbon/quartz materials are concerned, on the foundation of the same type of thermal protective results, masses are even lighter. The ratios of the masses of the three--carbon/quartz, molybdenum alloy, and coated molybdenum alloy are 1:1.62:1.70.

(3) Comprehensive Environmental Tests on Carbon/Quartz Large Dimension Structural Plates

In order to evaluate overall thermal and mechanical properties of materials under various types of environments, designs were done of thermal protective structural test items. The dimensions of carbon/quartz plates are 300mmx300mm--relatively close to dimensions among products which might be made use of. Other structural connections and insulation methods also approximate utilization situations. Fig.6 is a structural diagram of test items. Testing in accordance with flight programs, such experiments as room temperature vibration, room temperature shock, low temperature vibration, low temperature shock, as well as reentry heating, and so on, are gone through in sequence.

As far as the tests which are completed in sequence above are concerned, in actuality, they are comprehensive evaluation tests of radiant thermal protective structures, causing carbon/quartz to undergo in an integrated manner flight trials in various environments. The thermal effects and dynamic load effects all agree with design calculations. Carbon quartz plates also lack generation of such impermissible destruction as

cracking, resonance, low temperature brittleness fracture, high temperature oxidation, the pulling apart of connecting holes, and so on. As a result, tests clearly show that carbon/quartz plates are capable of complete replacement of refractory metals in making radiant skin with high thermal protective efficiencies.

Table 5 Small Plate Thermal Simulation Test Results

(1) 材 料	试件质量/g (2)	表面最高温度 (3) /°C	达到最高温时间 (4) /s	备 (5) 注
(6) 碳/石英	32.4	1 094	130	表面未处理，有烟 (9)
(6) 碳/石英	37.4	1 106	130	
(6) 碳/石英	31.9	1 041	148	
(6) 碳/石英	33.2	1 045	143	
(6) 碳/石英	29.2	1 057	134	
(6) 碳/石英	—	1 045	140	
(7) M_0板涂层	58	1 080	150	
(7) M_0板涂层	58	1 080	150	涂层有裂纹 (10)
(8) M_0—0.5Ti板	.55	1 182～1 215	126	涂层有裂纹 (10)

Key: (1) Materials (2) Test Sample Mass (3) Maximum Surface Temperatures (4) Time Reaching Maximum Temperature (5) Remarks (6) Carbon/Quartz (7) Mo Plate Coating (8) Mo-0.5 Ti Plate (9) Untreated Surfaces Show Smoke (10) Coatings Have Cracks

(4) Practical Applications of Carbon/Quartz Materials on Returnable Satellites

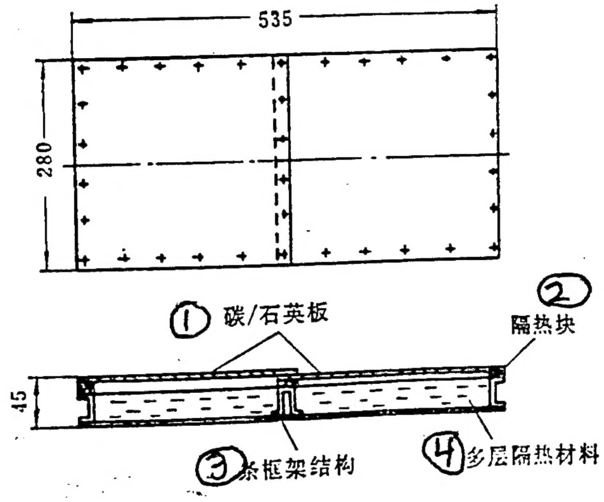

Fig.6 Structural Plate Test Sample

Key: (1) Carbon Quartz Plates (2) Insulation Block (3) Strip Framework Structure (4) Multiple Layer Insulation Material

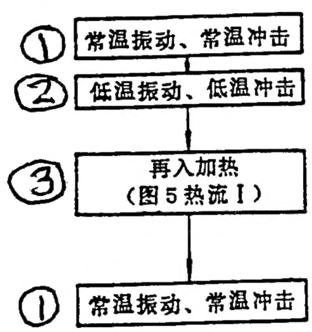

Fig.7 Test Cycle Flow Chart

Key: (1) Room Temperature Vibration Room Temperature Shock (2) Low Temperature Vibration Low Temperature Shock (3) Reentry Heating (Fig.5 Heat Flow I)

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Ever since 1975, carbon/quartz materials have already been utilized on over 20 Chinese returnable satellites for partial thermal protection. After 1984, reutilization of materials was also realized. Carbon/quartz materials--because of their possessing thermal expansion coefficients and rates of recession after ablation very close to quartz--are, therefore, unusually ideal materials for utilization as partial thermal protection in the vicinity of quartz material antenna windows. Approaching rates of thermal expansion for quartz, it is possible to avoid the application of thermal stresses to quartz windows during low temperatures associated with orbital sections. Synchronously with ablation, the appearance of tiering after burning corrosion of contact surfaces is, then, entirely avoided, thereby preventing the appearance of thermal flow interference.

3 CONCLUSIONS

Carbon fiber enhanced quartz is a high temperature inorganic composite material developed, and, in conjunction with that, first applied to satellite reentry thermal protective technologies by China. The material makes use of carbon fibers to carry out strengthening of quartz substrates--not only retaining the advantages of ceramic temperature resistance and insulation, but also overcoming the inherent brittleness of ceramics. In comparisons with other candidate radiant skin materials--for instance, high temperature alloys and refractory metals--it possesses such advantages as high temperature resistance, high specific strengths, strong insulation properties, and so on.

Before formal utilization in models, broad exploratory research was carried out aimed at thermal protective properties of materials and brittleness which may possibly be critical to utilization. In the experiments, typical flight environments associated with China's returnable type satellites were used as basis in the carrying out of room temperature vibration and shock, low temperature vibration and shock, as well as reentry heating tests on large scale structural members. Tests clearly demonstrated that carbon/quartz materials were completely suited to the various types of flight environments associated with China's returnable type satellites. In conjunction with this, they displayed very great potential for repeated utilization.

As far as exploratory research on carbon/quartz materials is concerned--as well as applications in returnable satellite partial thermal protective structures--for the sake of expanding

the use of this type of material to the thermal protective structures of manned spacecraft, a good deal of experience and data was accumulated. However, work requiring further expansion includes at least the following few items:

1) test measurement technologies associated with multiple items of thermophysical and mechanical properties in materials from room temperature to high temperature;

2) the influences of microscopic material structures such as fiber content and fiber direction on various properties;

3) nondestructive testing of stable forming techniques associated with thin plates having dimensions on the order of 1000mm as well as internal material quality;

4) preventive methods and testing technologies associated with effects on materials under high temperature conditions during vibration and shock;

5) prediction and repair techniques associated with the number of repeated utilizations.